BatchColumn Application Example

Solvent Regeneration

Example purpose
This example demonstrates the simulation of solvent regeneration using BatchColumn. A scenario is proposed to allow the separation of methanol, acetone, dichloromethane and diacetone alcohol.

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Corresponding BatchColumn files	*BATCHCOL_EX_EN-Solvent-regeneration.pbpc*

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1. Introduction

From the production of medicines to the manufacture of household rubber gloves, solvents play an essential role in modern society. However, they have one thing in common: the extensive use of solvents. The presence of solvents in the final products is negligible [SMA02]. It is therefore necessary to think about their regeneration to avoid their discharge or incineration. Batch distillation is one of the ways to do this.

Batch distillation is a frequently applied process in the fine chemical and pharmaceutical industry since it can be used to treat mixtures of variable quantity and composition. For batch distillation, several cuts can be taken, which is especially useful when a multicomponent waste solvent mixture is processed, since the components of these mixtures often form azeotropes with each other. The main component(s) to be recovered are obtained in the main cut(s), while the components and azeotropes having a lower or higher boiling point than the main component are removed in fore-cuts or after-cuts, respectively [NEM20]. In industry, it frequently occurs that a given amount of waste solvent must be processed in a given time interval; otherwise, the unprocessed amount is incinerated. Hence, increasing the processing capacity of waste solvent treatment might be crucial in order to make the whole production cleaner, which means that less waste is produced, more solvent is recovered and recycled, and the environmental footprint of the whole process is decreased [NEM20].

BatchColumn, Fives ProSim's batch distillation column simulation software, makes it possible to simulate solvent regeneration processes. Among other things, it makes it possible to adapt the scenario to the mixture to be treated, to optimize stop events to maximize the purity of the products of interest, to reduce inter-cuts, operating time, energy consumption, etc.

In this example, a mixture of methanol, acetone, dichloromethane, and diacetone alcohol is considered. Regeneration is carried out under reduced pressure: 100 mmHg. The table below shows, in ascending order of bubble temperatures, the 4 pure compounds and the two homogeneous azeotropes that methanol forms with acetone and dichloromethane.

Compound		T_{bubble} or $T_{azeotrope}$ (°C)	Molar fraction of compound 1	System type
1	2			
Methanol	Dichloromethane	-7.98	0.0754	Homogeneous
Dichloromethane	-	-7.14	-	-
Methanol	Acetone	7.16	0.0345	Homogeneous
Acetone	-	7.18	-	-
Methanol	-	20.63	-	-
Diacetone alcohol	-	107.15	-	-

From the previous table, it is proposed to divide the scenario into 5 parts in order to recover each solvent with an acceptable purity:

1) **Filling of the column**

   This step heats the initial load of the column from its storage temperature (0°C) to its boiling temperature and then fills the liquid hold-ups of the internals and the condenser.

2) **Dichloromethane recovery**

   The dichloromethane is recovered in a dedicated tank during a succession of distillation steps alternating with total reflux steps. The total reflux steps allow the column to be reclassified by lowering the heaviest to the bottom of the column.

3) **Inter-cut**

   A dedicated tank is used to finish the recovery of the methanol, dichloromethane and azeotrope methanol – acetone in order to obtain the maximum amount of acetone at the desired purity in the next part.

4) **Acetone recovery**

   The acetone is recovered in a single step in a dedicated tank.

5) **Methanol recovery**

   The methanol is recovered in a single step in a dedicated tank.

As the diacetone alcohol is the heaviest, it will be recovered in the boiler at the end of the operations.

2. COMPOUNDS

The compounds considered in the simulation are the following ones:

Name	Formula	CAS number[1]
Methanol	CH_4O	67-56-1
Acetone	C_3H_6O	67-64-1
Dichloromethane	CH_2Cl_2	75-09-2
Diacetone alcohol	$C6H_{12}O_2$	123-42-2

The compounds are extracted from the standard database of Simulis Thermodynamics, the calculation server of physico-chemical properties and phase equilibria used in BatchColumn. The physico-chemical properties stored in this database are taken from the DIPPR database [ROW24].

3. THERMODYNAMIC MODEL

Due to the pressure level in this process (100 mmHg) and the polar nature of the polar compounds, the NRTL thermodynamic profile [REN68] is chosen. This model is based on aa heterogeneous approach. This non-predictive model requires binary interaction parameters. Those taken into account are listed below.

Binaries	C_{ij}	C_{ji}	α_{ij}	C_{ij}^T	C_{ji}^T	α_{ij}^T
Methanol – Acetone	167.81	320.05	0.3	1.4923	-2.6727	0
Methanol – Dichloromethane	74.14	1517.35	0.4830	0	0	0
Methanol – Diacetone alcohol	454.65	477.58	0.3	-2.2325	1.0086	0
Acetone – Dichloromethane	-725.20	641.70	0.3500	0	0	0
Acetone – Diacetone alcohol	2127.96	-1624.17	0.2908	0	0	0
Dichloromethane – Diacetone alcohol	1892.81	-765.46	0.3	-21.796	10.905	0

[1] *CAS Registry Numbers® are the intellectual property of the American Chemical Society and are used by Fives ProSim SAS with the express permission of ACS. CAS Registry Numbers® have not been verified by ACS and may be inaccurate.*

4. SIMULATION

4.1. Process description

4.1.1. Column

The column is made up of 32 theoretical stages, including the boiler and the condenser.

Four collection tanks will be used during the distillation step.

The initial load is detailed below:

✓ Temperature	0°C	
✓ Total mass	6 090 kg	
✓ Composition	Methanol	19.45% wt.
	Acetone	21.96% wt.
	Dichloromethane	56.29% wt.
	Diacetone alcohol	2.30% wt.

The liquid hold-ups are supposed to be constant all along the distillation:

- ✓ Condenser 15 l
- ✓ Column 2.5 l for each theoretical stage

The pressure profile is also supposed to be constant all along the distillation process

- ✓ Condenser 100 mmHg
- ✓ Pressure drop 10 mmHg

The stages efficiency is assumed to be equal to 1 (each stage is assumed to be a theoretical stage).

4.1.2. Condenser

The condenser is assumed to be ideal total all along the distillation process.

4.2. Operating mode

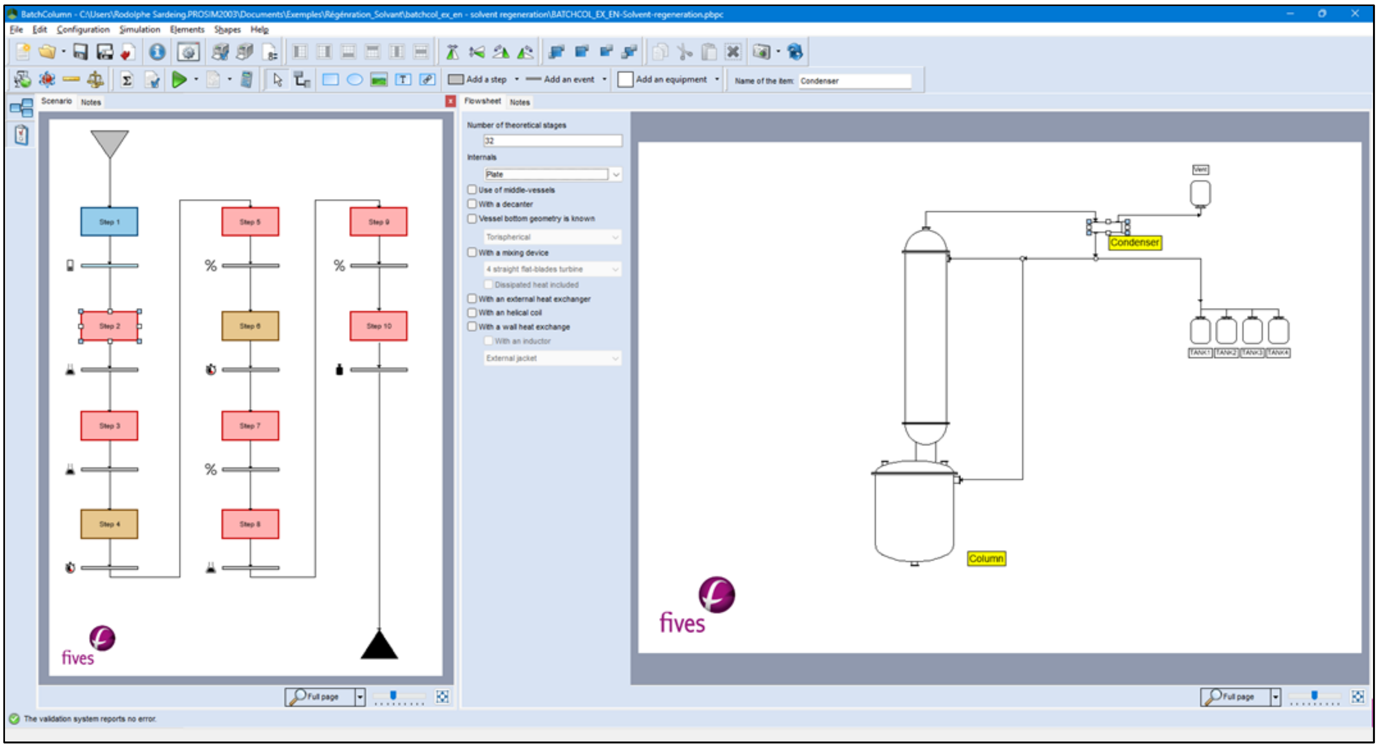
The operating mode consists of ten steps. The operating parameters of these steps are detailed in the tables below. The following settings are the same for all steps:

- ✓ Operating mode : Constant heat duty
- ✓ Biler heat duty : 500 000 kcal/h
- ✓ Condenser : Ideal total

Parameter	**Step 1**	**Step 2**	**Step 3**	**Step 4**	**Step 5**
	Filling	**Dichloromethane recovery**			
Type	Filling	Distillation		Infinite reflux	Distillation
Reflux ratio	-	2	5	-	5
Collection tank	-	Tank 1		-	Tank 1
Stop event	Column is filled	Total production inside tank 1 = 2 000 kg	Total production inside tank 1 = 2 800 kg	Step duration = 30 min	Dichloromethane mass fraction inside tank 1 < 0.96

Parameter	**Step 6**	**Step 7**	**Step 8**	**Step 9**	**Step 10**
	Dichloromethane recovery		**Inter-cut**	**Acetone recovery**	**Methanol recovery**
Type	Infinite reflux	Distillation			
Reflux ratio	-	5			
Collection tank	-	Tank 1	Tank 2	Tank 3	Tank 4
Stop event	Step duration = 30 min	Dichloromethane mass fraction inside tank 1 < 0.95	Total production inside tank 2 = 560 kg	Acetone mass fraction inside tank 3 < 0.9	Total load inside the boiler < 150 kg

The scenario is presented on the left of the following screenshot and the flowsheet on the right part.

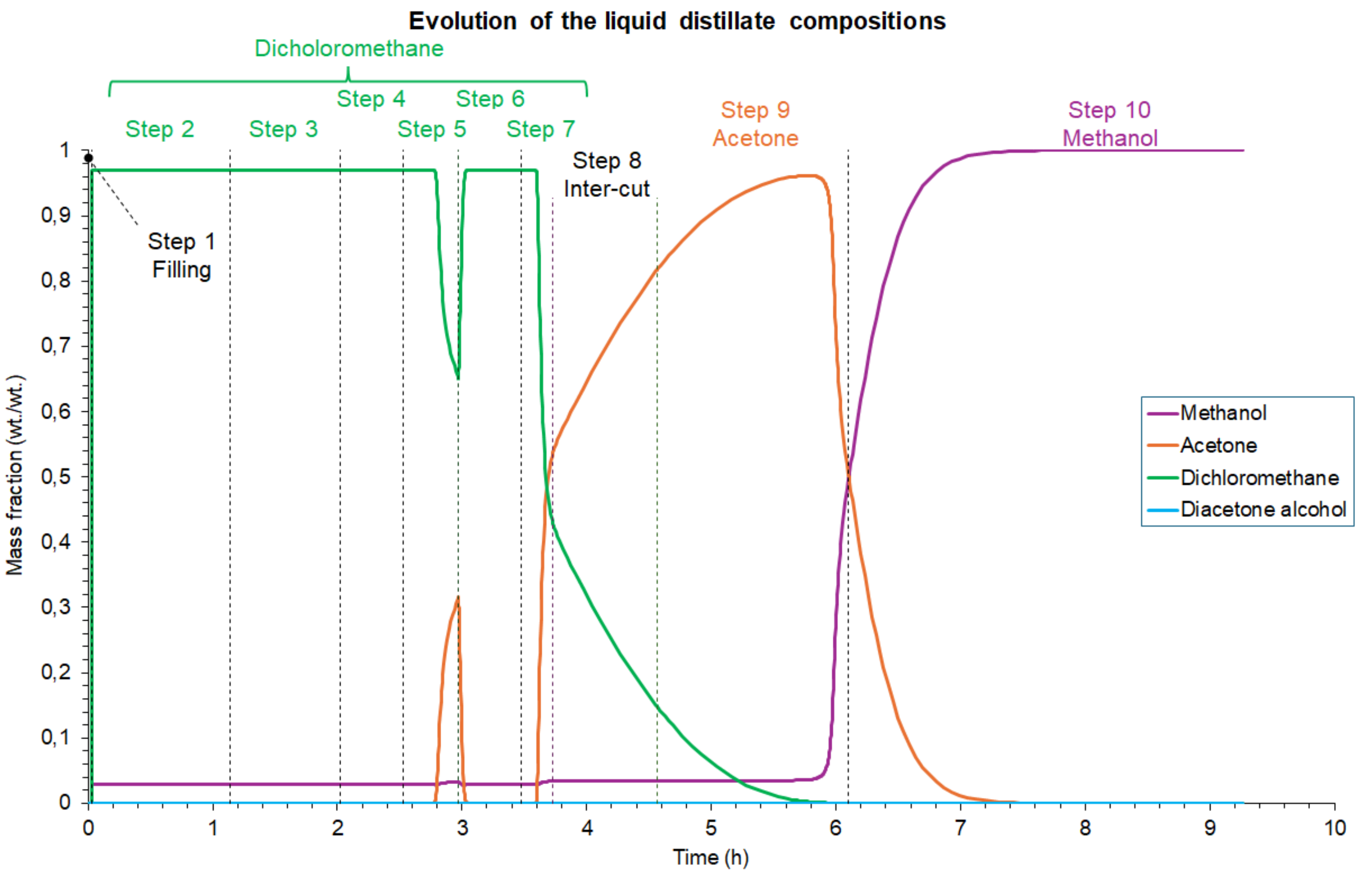


5. RESULTS

5.1. Profiles

Several profiles are available at the end of the simulation (temperatures, liquid distillate and boiler compositions, tank compositions, etc.), including the figure below. This figure shows the evolution of the mass fractions in the liquid distillate. Solvents are going to the head of the columns as the scenario evolves according to their bubble temperature. These profiles allow to visualize, among other things:

- ✓ The effect of the total reflux of the step 6 which allows to increase the dichloromethane content after it decreases at the end of the step 5.
- ✓ The effect of the inter-cut of step 8 which accelerates the decease of dichloromethane and the increase of acetone.



5.2. Tabulated results

The Word report generated at the end of the simulation contains summary tables, including the table below. This table shows the contents of the collection tanks, the boiler and the liquid hold-ups (condenser and internals) at the end of the operation. This table shows that the different solvents are properly separated in their dedicated tank.

	Tank 1	**Tank 2**	**Tank 3**	**Tank 4**	**Boiler**	**Liquid hold-up**
Cut	Dichloromethane	Inter-cut	Acetone	Methanol	Diacetone alcohol	-
Wight (kg)	3 403	560	926	980	150	71
Composition (weight %)						
Methanol	3.00	3.45	5.71	94.83	6.50	99.23
Acetone	2.00	68.69	90.00	5.17	0	0
Dichloromethane	95.00	27.86	4.29	0	0	0
Diacetone alcohol	0	0	0	0	93.50	0.77

6. REFERENCE

[NEM20] NEMETH B., LANG P., HEGELY L., “Optimisation of Solvent Recovery in Two Batch Distillation Columns of Different Size”, J. Cleaner Production, 275, 122746 (2020).

[REN68] RENON H., J.M. PRAUSNITZ, “Local Compositions in Thermodynamic Excess Functions for Liquid Mixtures”, AIChE J., 14(3), 135-144 (1968).

[ROW24] ROWLEY R.L., WILDING W.V., OSCARSON J.L., GILES N.F., “DIPPR® Data Compilation of Pure Chemical Properties”, Design Institute for Physical Properties, AIChE (2023).

[SMA02] SMALLWOOD I.M., “Solvent Recovery Handbook”, 2nd ed., CRC Press (2002).

